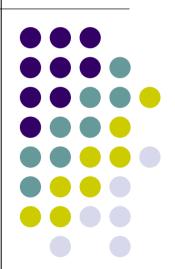
## Automated Analytical Laboratory

Eric Byrd Polymeric Materials Group May 17-18, 2006





#### **Background**



- High flux UV exposure studies on the NIST SPHERE result in short exposure times and rapid sample turnover.
- Research needs:
  - Automated and rapid analysis of specimens under study
  - Storage and processing of massive amounts of analytical data
  - Parallel measurement systems
- No satisfactory commercial solution exists for either of these needs.



#### **Background**

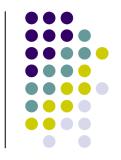


A comprehensive high throughput research program includes:

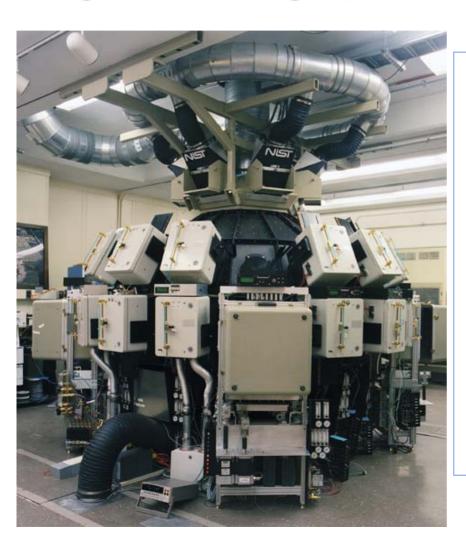
- Combinatorial methods for making specimens
- Accelerated aging process [NIST SPHERE]
- High throughput specimen characterization
- Informatics



#### **High Throughput UV Exposure**



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#### For one experiment:

32 ports

17 specimens/port

Typically, 6 different types of measurements/experiment

Typically, 50 measurement intervals/experiment

> 160,000 <u>measurements per</u> experiment!

(x4 for replicates)

# High Throughput UV Exposure Current Methodology



#### REMOVE SPECIMENS



UV-VISIBLE (Samples & Filters)



FTIR (Samples)



REPLACE SPECIMENS



4 Specimens

~ 6 hours

FTIR: 45 min

UV-Vis: 20 min



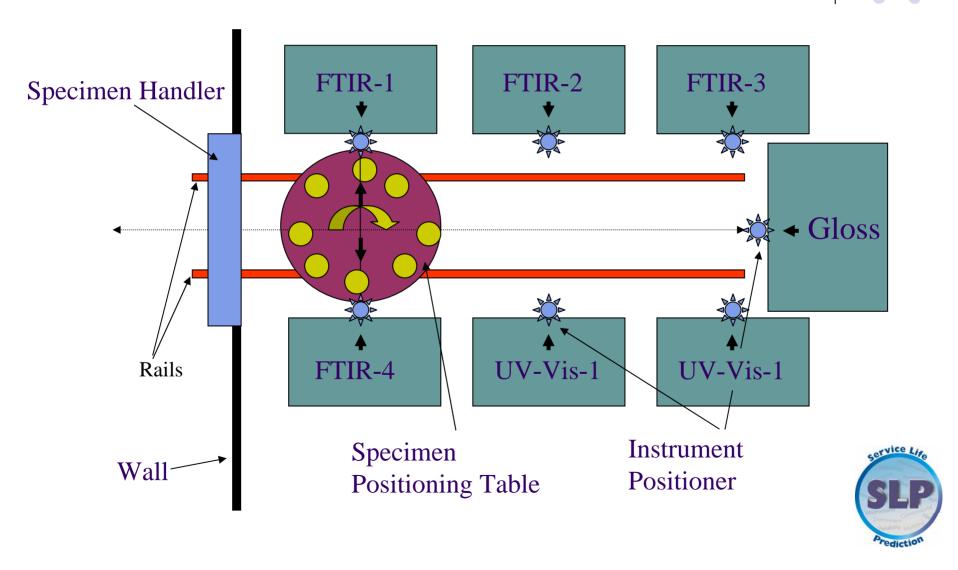
#### Goals



- Decrease specimen measurement time by a factor of at least 20 without sacrificing data integrity
- Minimize specimen handling to decrease errors.
- Standardize calibration and measurement protocols over multiple instrument platforms.
- Ensure enough flexibility and capacity for future needs.

## al

#### **Automated Analytical Design Proposal**



## Specimen Handling & Analytical Instruments



- Specimen Handling
  - 7-Axis Robot
  - Specimen Handler
  - Instrument Positioners
- Analytical Instruments
  - FTIR
  - UV-Vis
  - Glossmeter



#### 7-Axis Robot







### 7-Axis Robot Specifications

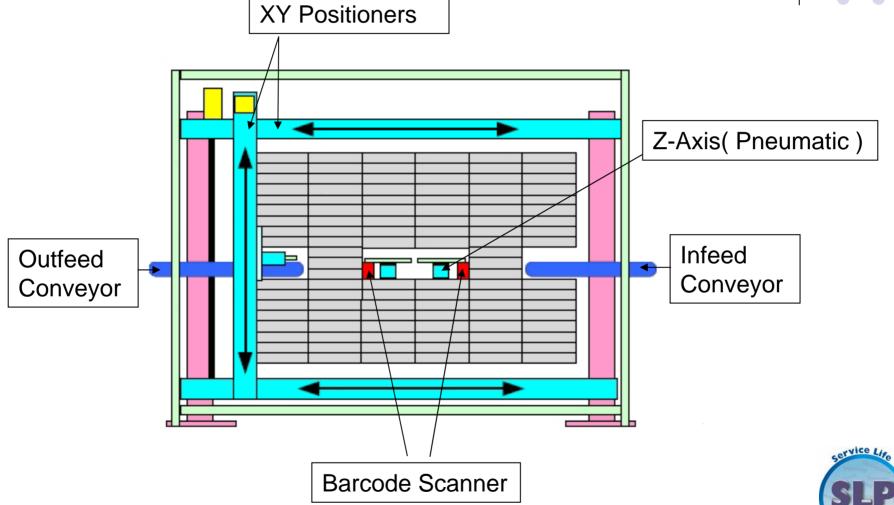


- Designed for laboratory use
- Seven degrees of freedom
  - 4 meter track
- Absolute encoders, no homing
- Payload: 3kg
- Reach (std. gripper): 863 mm
- Repeatability: +/- 0.05mm
- Weight 53 kg



#### **Proposed Specimen Handler**

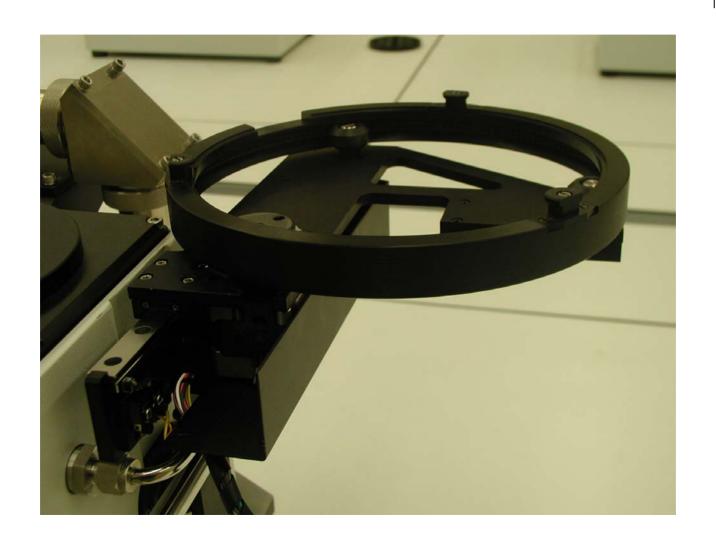




#### Instrument positioners

radius-theta positioner







#### Instruments



- FTIR
- UV-Vis
- Glossmeter

- Instrument assemblies (instruments + positioners) will be interchangeable.
  - Easily removable for maintenance
  - Modular programming
  - All components have a small "footprint"



#### MIDAC Illuminator FTIR





#### Interferometer

Type: 90° Michelson with duplexed mechanical

bearings and linear force motor

Mirror Control: HeNe laser with quadrature phase detectors

Mirrors: Gold coated, diamond turned, permanently

aligned

IR Source: 1650° K, air-cooled, permanently aligned Resolution: 0.5 or 1 cm-1, (step adjustable to 32 cm-1) Optics: KBr, ZnSe, or CaF2 beamsplitter and windows

Abscissa: 7800-350 cm-1 for KBr

Accuracy: >0.01 cm-1

Detector: DTGS or LN2 cooled MCT or InSb

(separate external module)

Dimensions: 8" x 14" X 8" (W x D x H)

www.midac.com



# MIDAC Illuminator FTIR with Custom Optical Interface

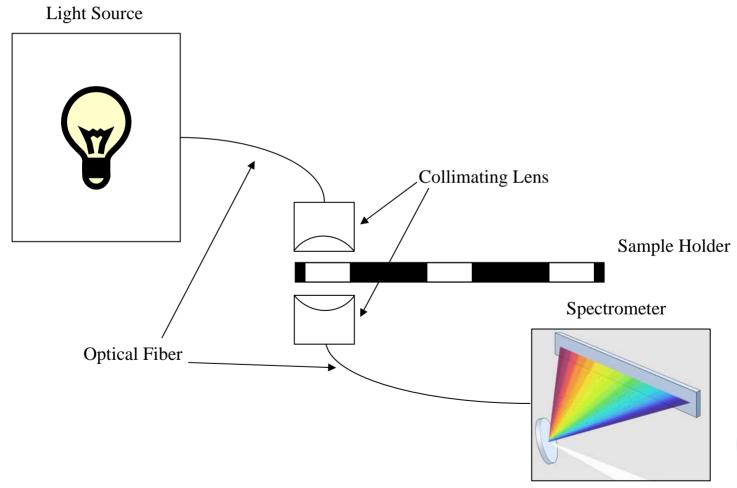






#### **Schematic of UV-Vis**

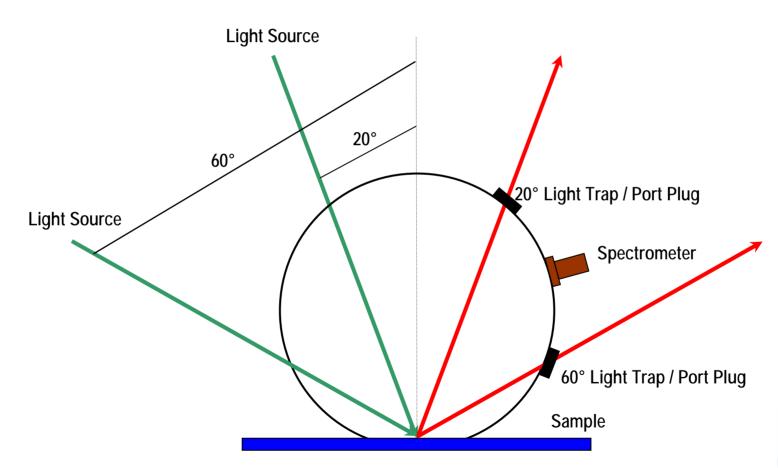






#### 20°/60° Glossmeter

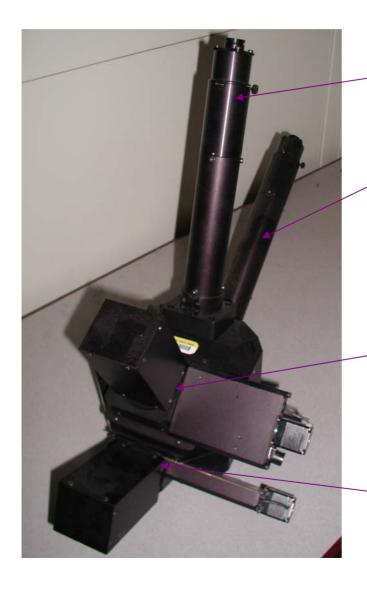






#### 20°/60° Glossmeter





60° Light Source

20° Light Source

60° Port Plug/Light Trap

20° Port Plug/Light Trap



#### **Calibration**



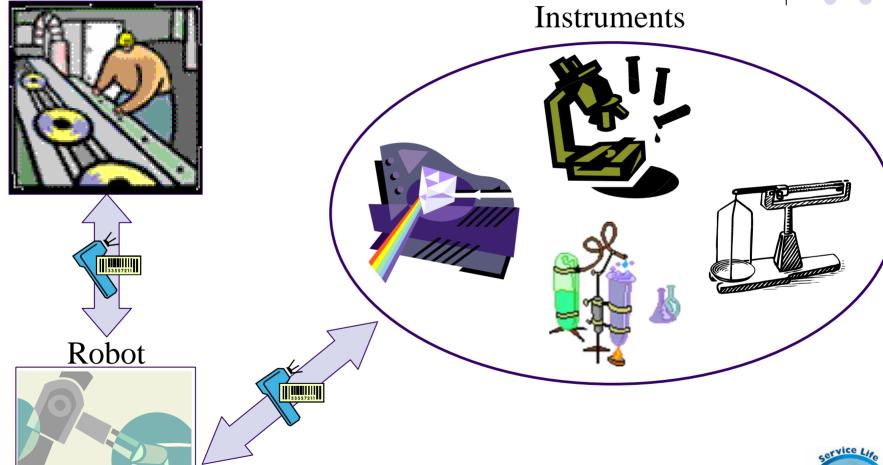
- NIST Standard Reference Material (SRM)
  - UV-Vis
  - Glossmeter
  - FTIR

Calibrations will be performed routinely



### Sampling methodology

Specimen Handler







#### **Additional Instruments**



- FTIR ATR
- UV-Vis / NIR Reflection Spectrometer
- Raman Spectrometer
- Fluorescence Spectrometer
- Microscope



#### **Gantt chart**



Y1 Y2 Y3 Y4

7-Axis Robot, FTIR-T, Glossmeter (completed)

UV-Vis Spectrometers Rack-Mount Computers Sample Handler Instrument Positioners

Investigate/Purchase instruments for pigmented samples

Implement new instruments



# **Automated Analytical Laboratory**





